



PATENT APPLICATION  
Mo-6599  
Lea 34,920

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF )  
WERNER OBRECHT ET AL ) GROUP NO.: 1712  
SERIAL NUMBER: 10/033,247 ) EXAMINER: P. A. SHORT  
FILED: DECEMBER 29, 2001 ) RESPONSE TO PAPER # 1003  
TITLE: MICROGEL-CONTAINING RUBBER )  
COMPOUNDS WITH PHOSPHORYL )  
POLYSULFIDES AND )  
VULCANIZATES OR SHAPED )  
ARTICLES PREPARED THEREFROM)

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents

P. O. Box 1450

Alexandria, VA 22313-1450

Sir:

I, Dr. Thomas Früh, of Limburgerhof, Germany declare as follows:

1. I studied chemistry at **Universität Hannover** and obtained a **Dr. in 1996**.
2. I am employed by **Rhein Chemie Rheinau GmbH**, in Mannheim Germany as a **Manager R&D**.
3. I am one of the named inventors of the above-identified United States patent application.
4. I performed or supervised the experiments described in the above-identified United States patent application.

5. I performed or supervised the following experiment:

a. Compound 6 was prepared according to the following formulation (Comparative Table 1) in a closed laboratory mixer under the same conditions as Compounds 1-5 were prepared in the above identified United States patent application.

Comparative Table 1:

Compound No:	6
TSR 5, Defo 700 <sup>1)</sup>	25
NR master batch with 50 wt.% of SBR microgel	150
Carbon black N121	0
Antiozonant wax <sup>2)</sup>	1.5
Stearic acid	3
Zinc oxide	3
IPPD <sup>3)</sup>	1
TMQ <sup>4)</sup>	1
Sulfur	1.6
TBBS <sup>5)</sup>	1
Zinc phosphoryl polysulfide <sup>6)</sup>	0
phosphoryl polysulfide <sup>7)</sup>	4

1) = TSR 5, Defo 700 (masticated natural rubber with Defo hardness 700)

2) = Antilux L® from Rheinchemie

3) = N-isopropyl-N'-phenyl-p-phenylenediamine (Vulcanox® 4010 from Bayer AG)

4) = 2,2,4-trimethyl-1,2-dihydroquinoline/polym. (Vulcanox® HS from Bayer AG)

5) = N-tert.butyl-2-benzothiazylsulfenamide (Vulkacit NZ from Bayer AG)

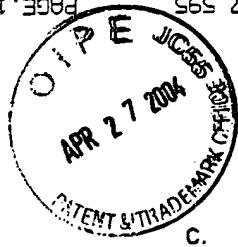
6) = zinc bis(O,O-2-ethylhexyl)dithiophosphate (Rhenocure® ZDT/G/50% strength)

7) = bis(O,O-2-ethylhexylthiophosphoryl)polysulfide (Rhenocure® SDT/G/50% strength)

b. The viscosity of Compounds 1-5, as filed in the above identified patent application and Compound 6 are collated in the following table:

Comparative Table 2:

Compound No:	1	2	3	4	5	6
Compound viscosity/ML 1+4 [ME]	59.7	53.5	49.9	55	60.5	51.05



c. Characteristic vulcanization times for Compound 6, for example  $t_{10}$ ,  $t_{50}$ ,  $t_{90}$ ,  $t_{95}$  and characteristic torques such as  $S_{max}$  and  $S_{min}$ , were determined in a rheometer experiment at 160°C using a Monsanto Rheometer 100 as Compounds 1-5 in the above identified patent application.

Comparative Table 3:

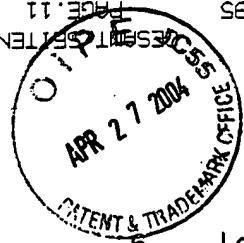
Compound No:	1	2	3	4	5	6
$t_{50}$ [min]	4.8	3.2	2.8	1.7	1.9	2.4
$t_{10}$ [min]	5	3.3	3	1.9	2.1	2.3
$t_{50}$ [min]	7.7	4.4	4.2	2.5	2.8	3.3
$t_{90}$ [min]	11.5	7.1	6.7	4.6	5	5.0
$t_{95}$ [min]	13.4	8.6	8.2	5.3	6.1	6.0
$S_{max}$ [Nm]	13.7	14.5	15.6	17.3	19.0	13.5
$S_{min}$ [Nm]	3.0	2.8	2.6	3.2	3.0	2.8

d. Compound 6 was vulcanized for 9 minutes at 160°C in a press, as Compounds 1-5 were in the above identified patent application.

The following test results are obtained:

Comparative Table 4

Compound No:	1	2	3	4	5	6
Tensile strength [MPa]	18.9	19.0	18.5	18.2	20.8	21.4
elongation at break (D) [%]	485	465	430	510	400	498
modulus at 100% elongation ( $S_{100}$ ) [MPa]	1.8	1.7	2.0	1.9	2.6	1.6
modulus at 300% elongation ( $S_{300}$ ) [MPa]	7.5	8.4	9.7	11.5	13.6	8.0
Shore A hardness, 23°C	61	61	62	63	67	58
Shore A hardness, 70°C	54	54	56	58	61	53
Resilience, 23°C [%]	35	37	38	35	36	40
Resilience, 70°C [%]	60	63	65	59	60	64
Abrasion 60th emery [ $\text{mm}^3$ ]	140	155	130	142	135	144
$S_{300} \times D$	3638	3906	4171	5865	5440	3984



6. I declare that one skilled in the art would recognize that use of phosphoryl polysulfide in a SBR/NR masterbatch as claimed in the present invention provides a compound with improved properties, specifically increased tensile strength, elongation a break (D) and in  $S_{300} \times D$  when compared to a compound made in the absence of phosphoryl polysulfide.

7. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the the above-identified United States patent application or any patent issuing therefrom.

Signed at Mannheim Germany, this 16<sup>th</sup> day of April, 2004.

A handwritten signature in black ink, appearing to read "Früh".

Dr. Thomas Früh

lo/SENG/jrs214